4. By adding Reference Method 21 to Appendix A as follows:

Appendix A—Reference methods

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Method 21. Determination of Volatile Organic Compound Leaks

1. *Applicability and Principle*

1.1 Applicability. This method applies to the determination of volatile organic compound (VOC) leaks from organic process equipment. These sources include, but are not limited to, valves, flanges and other connections, pumps and compressors, pressure relief devices, process drains, open-ended valves, pump and compressor seal system degassing vents, accumulator vessel vents, and access door seals.

1.2 Principle. A portable instrument is used to detect VOC leaks from individual sources. The instrument detector is not specified, but it must meet the specifications and performance criteria contained in paragraph 2.1.

2. *Apparatus*

2.1 Monitoring Instrument. The monitoring instrument shall be as follows:

2.1.1 Specifications.

a. The VOC instrument detector shall respond to the organic compounds being processed. Detectors which may meet this requirement include, but are not limited to, catalytic oxidation, flame ionization, infrared absorption, and photoionization.

b. The instrument shall be intrinsically safe for operation in explosive atmospheres as defined by the applicable U.S.A Standards (e.g., [illegible] Code by the National Fire Prevention Association).

c. The instrument shall be able to measure the leak definition concentration specified in the regulation.

d. The instrument shall be equipped with a pump so that a continuous sample is provided to the detector. The nominal sample flow rate shall be 1–3 liters per minute.

e. The scale of the instrument meter shall be readable to ±5 percent of the specified leak definition concentration.

2.1.2 Performance Criteria. The instrument must meet the following performance criteria. The definitions and evaluation procedures for each parameter are given in Section 4.

2.1.2.1 The instrument response time must be 30 seconds or less. The response time must be determined for the instrument system configuration to be used during testing, including dilution equipment. The use of a system with a shorter response time than that specified will reduce the time required for field component surveys.

2.1.2.2 Calibration Precision: The calibration precision must be less than or equal to 10 percent of the calibration gas value.

2.1.2.3 Quality Assurance. The instrument shall be subjected to response time and calibration precision tests prior to being [illegible] service. The calibration precision [illegible] be repeated every 6 months [illegible] modification or replacement [illegible] detector is required, the [illegible] be retested and a new 6 [illegible] assurance test schedule will apply. The response time test shall be repeated if any modifications to the sample pumping system or flow configuration is made that would change the response time.

2.3 Calibration Gases. The monitoring instrument is calibrated in terms of parts per million by volume (ppmv) of the compound specified in the applicable regulation. The calibration gases required for monitoring and instrument performance evaluation are a zero gas (air, 3 ppmv VOC) and a calibration gas in air mixture approximately equal to the leak definition specified in the regulation. If cylinder calibration gas mixtures are used, they must be analyzed and certified by the manufacturer to be within ±2 percent accuracy. Calibration gases may be prepared by the user according to any accepted gaseous standards preparation procedure that will yield a mixture accurate to within ±2 percent. Alternative calibration gas species may be used in place of the calibration compound if a relative response factor for each instrument is determined so that calibrations with the alternative species may be expressed as calibration compound equivalents on the meter readout.

3. *Procedures*

3.1 Calibration. Assemble and start up the VOC analyzer and recorder according to the manufacturer's instructions. After the appropriate warmup period and zero or internal calibration procedure, introduce the calibration gas into the instrument sample probe. Adjust the instrument meter readout to correspond to the calibration gas value.

If a dilution apparatus is used, calibration must include the instrument and dilution apparatus assembly. The nominal dilution factor may be used to establish a scale factor for converting to an undiluted basis. For example, if a nominal 10:1 dilution apparatus is used, the meter reading for a 10,000 ppm calibration would be set at 1,000. During field surveys, the scale factor of 10 would be used to convert measurements to an undiluted basis.

3.2 Individual Source Surveys.

3.2.1 Case I—Leak Definition Based on Concentration Value. Place the probe inlet at the surface of the component interface where leakage could occur. Move the probe along the interface periphery while observing the instrument readout. If an increased meter reading is observed, slowly probe the interface where leakage is indicated until the maximum meter reading is obtained. Leave the probe inlet at this maximum reading location for approximately two times the instrument response time. If the maximum observed meter reading is greater than the leak definition in the applicable regulation, record and report the result as specified in the regulation reporting requirements. Examples of the application of this general technique to specific equipment types are:

a. Valves—The most common source of leaks from valves is at the seal between the stem and housing. Place the probe at the interface where the stem exists the packing gland and sample the stem circumference. Also, place the probe at the interface of the packing gland take-up flange seat and sample the periphery. In addition, survey valve housings of multipart assembly at the surface of all interfaces where leaks can occur.

b. Flanges and Other Connections—For welded flanges, places the probe at the outer edge of the flange-gasket interface and sample around the circumference of the flange. Sample other types of nonpermanent joints (such as threaded connections) with a similar traverse.

c. Pumps and Compressors—Conduct a circumferential traverse at the outer surface of the pump or compressor shaft and seal interface. If the source is a rotating shaft, position the probe inlet within one centimeter of the shaft-seal interface for the survey. If the housing configuration prevents a complete traverse of the shaft periphery, sample all accessible portions. Sample all other joints on the pump or compressor housing where leakage can occur.

d. Pressure Relief Devices—The configuration of most pressure relief devices prevents sampling at the sealing seat interface. For those devices equipped with an enclosed extension, or horn, place the probe inlet at approximately the center of the exhaust area to the atmosphere for sampling.

e. Process Drains—For open drains, place the probe inlet at approximately the center of the area open to the atmosphere for sampling. For covered drains, place the probe at the surface of the cover interface and conduct a peripheral traverse.

f. Open-Ended Lines or Valves—Place the probe inlet at approximately the center of the opening to the atmosphere for sampling.

g. Seal System Degassing Vents and Accumulator Vents—Place the probe inlet at approximately the center of the opening to the atmosphere for sampling.

h. Assess Door Seals—Place the probe inlet at the surface of the door seal interface and conduct a peripheral traverse.

3.2.2. Case II-Leak Devinition Based on "NO Detactable Emission".

a. Determine the local ambient concentration around the source by moving the probe inlet randomly upwind and downwind at distance of one to two meters from the source. If an interference exists with this determination due to a nearby emission or leak, the local ambient concentration may be determined at distances closer to the source, but in no case shall the distance be less than 25 centimeters. Note the ambient concentration and then move the probe inlet to the surface of the source and conduct a survey as described in 3.2.1. If a concentration increase greater than 2 percent of the concentration-based leak definition is obtained, record and report the results as specified by the regulation.

b. For those cases where the regulation requires a specific device installation, or that specified vents be ducted or piped to a control device, the existence of these conditions shall be visually confirmed. When the regulation also requires that no detectable emissions exist, visual observations and sampling surveys are required. Examples of this technique are:

i. Pump or Compressor Seals—If applicable, determine the type of shaft seal. Perform a survey of the local area ambient VOC concentration and determine if detectable emissions exist as described in 3.2.2.a.

ii. Seal system degassing vents, accumulator vessel vents, pressure relief

devices—If applicable, observe whether or not the applicable ducting or piping exists. Also, determine if any sources exist in the ducting or piping where emissions could occur prior to the control device. If the required ducting or piping exists and there are no sources of where the emissions could be vented to the atmosphere prior to the control device, then it is presumed that no detectable emissions are present.

4. Instrument Performance Evaluation Procedures

4.1 Definitions.

4.1.1 Calibration Precision. The difference between the average VOC concentration indicated by the meter readout for consecutive repetitions and the know concentration of a test gas mixture.

4.1.2 Response time. The time interval from a step change in VOC concentration at the input of the sampling system to the time at which 90 percent of the corresponding final value is reached as displayed on the instrument readout meter.

4.2 Evaluation Procedures. At the beginning of the instrument performance evaluation test, assemble and start up the instrument according to the manufacturer's instructions for recommended warmup period and preliminary adjustments. If a dilution apparatus is used during field surveys, the evaluation procedure must be performed on the instrument-dilution system combination.

4.2.1. *Calibration Precision Test.* Make a total of nine measurements by alternately using zero gas and the specified calibration gas. Record the meter readings (example data sheet shown in Figure 21–1).

4.2.2 Response time Test Procedure. Introduce zero gas into the instrument sample probe. When the meter reading has stabilized, switch quickly to the specified calibration gas. Measure the time from concentration switching to 95 percent of final stable reading. Perform this test sequence three times and record the results (example data sheet given in Figure 21–2).

4.3 Calculations. All results are expressed as mean values, calculated by:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i$$

Where:

x_i = VALUE OF THE MEASUREMENTS.

Σ = Sum of the individual values.

$\bar{x}$ = Mean value.

n = Number of data points.

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